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Its Emergence and Expansion

Springer

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Anthony S. Travis
Sidney M. Edelstein Center for the History
and Philosophy of Science,
Technology and Medicine
The Hebrew University of Jerusalem
Jerusalem
Israel

ISSN 2191-5407 ISSN 2191-5415 (electronic)
SpringerBriefs in Molecular Science
ISSN 2212-991X
SpringerBriefs in History of Chemistry
ISBN 978-3-319-19356-4 ISBN 978-3-319-19357-1 (eBook)
DOI 10.1007/978-3-319-19357-1

Library of Congress Control Number: 2015940967

Springer Cham Heidelberg New York Dordrecht London

Printed on acid-free paper

Springer International Publishing AG Switzerland is part of Springer Science+Business Media
(www.springer.com)

Preface

In 1909, the German physical chemist Fritz Haber devised an apparatus for the synthesis of ammonia from its elements, hydrogen and nitrogen, under conditions of very high pressure and temperature, in the presence of a catalyst, osmium in particular. Four years later, in September 1913, mainly thanks to the industrial chemist Carl Bosch at BASF, Haber's method was turned into a process for the manufacture of nitrogen fertilizer. It was, arguably, one of the greatest inventions of the twentieth century, and for many certainly the most beneficial. However, the inauguration less than a year before the outbreak of World War I of what became known as the Haber-Bosch process would mean that its earliest large-scale application was to be in the manufacture of explosives for the Kaiser's armed forces. Nevertheless, and despite much belief to the contrary, then and now, it was not the only important process employed in Germany for the manufacture of nitrogen products during the war.

My own interest in nitrogen products began in the early 1980s, when working with Brent Schools and Industry Project, a programme of the London Borough of Brent aimed at introducing the application of science into the classroom. At that time, the few studies of the history of the ammonia process and of related early twentieth-century nitrogen fixation processes were written for specialist audiences. The outcome of my research was the publication in 1984 of *The High Pressure Chemists*. Usefully, the project was aided by the presence in Brent, at North Wembley, of the research laboratories of the (British) General Electric Company (GEC), with which Haber's skilful coinventor Robert Le Rossignol was associated. It was through former GEC head chemist the late Dr. Ralph C. Chirnside, a close friend of Le Rossignol, that I learnt about some of the connections with Haber. No less important was the manufacturing and research facility of Johnson Matthey Metals Limited located on the site of the former Wembley Exhibition grounds. There I was able to view the weaving of platinum wire gauzes required in the oxidation of ammonia to nitric acid, a vital step in the munition manufacture that was brought to perfection under conditions of war after 1914. Johnson Matthey also operated a nitric acid pilot plant adjacent to the facility.

Since the 1980s, there have appeared several published accounts of the life and work of Fritz Haber, who also happens to be associated with the introduction of large-scale gas warfare in 1915. None of these accounts, however, deal in a balanced way with the technical story of both ammonia, with which Haber and Carl Bosch were so intimately associated, and the rival nitrogen processes, in particular the electric arc and Frank–Caro (cyanamide) processes. It is in order to make up for this lacuna that I here present the result of an extensive reworking of my earlier research, incorporating the studies of several colleagues, including participants in the European Science Foundation's Evolution of Chemistry in Europe, 1789–1939 programme. I would especially like to acknowledge the members of the committee of the Historical Group of the Royal Society of Chemistry who kindly invited me to give the 2014 Wheeler Award Lecture on the topics dealt with here. The staff of the Wellcome Collection, London, and the Sidney M. Edelstein Library for the History and Philosophy of Science, Technology and Medicine at the National Library of Israel, Jerusalem, are thanked for great assistance. Luca Bianchi of Casale SA kindly answered a number of questions and provided useful background information. A special thanks to Peter J. T. Morris, of the Science Museum, London, with whom I have shared an interest in the history of chemical technology for over two decades, and at whose suggestion I undertook the writing of this monograph. I am grateful to Dr. Morris for extensive review of an earlier version of the manuscript and for kindly providing information based on his own research. Finally, it is important to emphasize that the events described in Chaps. 1 and 2 are as much a prelude to World War I as were the stories of the build-up of fleets of battleships among the "Great Powers" in the decade or so prior to 1914. In Chaps. 4 and 5, this is reflected, at the climax of the war in November 1918, through the vast nitrogen factories undergoing expansion, under construction or planned in Germany, Britain, France, and the United States, and, in the aftermath, the struggles until the mid-1920s to develop rivals to the Haber-Bosch process mainly based on research started during 1916–1918.

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Units of measurement are as given in the original sources.

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About the Author

Anthony S. Travis Ph.D. is deputy director of the Sidney M. Edelstein Center for the History and Philosophy of Science, Technology and Medicine at The Hebrew University of Jerusalem (tony.travis282@gmail.com). He has published extensively on the history of chemistry and chemical technology in the nineteenth and twentieth centuries. Travis is the recipient of four awards for his contributions to the history of chemistry.

He has edited and coedited several volumes and special issues of journals dealing with the history of chemical sciences, technology and the environment. Publications include *The Rainbow Makers: The Origins of the Synthetic Dyestuffs Industry in Western Europe* (Lehigh: Lehigh University Press, 1993); (with Carsten Reinhardt) *Heinrich Caro and the Creation of Modern Chemical Industry* (Dordrecht: Kluwer, 2000); *Dyes Made in America, 1915–1980: The Calco Chemical Company, American Cyanamid, and the Raritan River* (Jerusalem: Edelstein Center/Hexagon Press, 2004); and *On Chariots with Horses of Iron and Fire: The Excursionists and the Narrow Gauge Railroad from Jaffa to Jerusalem* (Jerusalem: Magnes Hebrew University Press/Shapell Manuscript Foundation, 2009).

Chapter 1
Introduction

1.1 War and Fixed Nitrogen

> The story still is told of a Minister, a member of the War Cabinet, who, finding the conversation at a certain dinner turning to the sinister menace of the submarine campaign, then at its height, and its effects especially on the Chile communications, turned to his neighbour with the enquiry: 'Tell me, what *is* this nitrate they are all making such a fuss about?'
>
> Stanley I. Levy, "The Status of Chemists and Chemistry," in *Chemistry and Industry*, no. 11, 14 March 1924, pp. 285–286.

Apocryphal or not, this extract from the correspondence columns of the then new British journal *Chemistry and Industry* in 1924 exposes the apparent general ignorance in Britain, and also for a time in Germany, of a crucial and often desperate episode in the conduct of World War I. "Nitrate," a commodity essential to the production of all modern explosives used in warfare, mainly the aromatic nitro compounds such as TNT (trinitrotoluene) and picric acid, was common currency to all belligerents. Nevertheless outside of scientific and industrial circles the critical roles of what was in fact Chilean nitrate (Chile saltpetre, or sodium nitrate; the term saltpetre refers to the potassium salt), and other nitrogen-containing chemicals of commerce, such calcium cyanamide and ammonia, as sources of vast destructive power, was generally given little, if any, prominence at the start of the war in early August 1914.

Within weeks the German Army, pursuing the campaign drawn up by Count Alfred von Schlieffen, had occupied Luxembourg, crossed neutral Belgium, and entered northern France, laying waste to towns and cities. Acts of aggression were reported, and sometimes greatly exaggerated. Belgian civilians were shot, including in groups, in response to trumped up charges of sabotage, and the ancient library at Louvain was reduced to ashes. In France, Lille and Douai were lost, the cathedral at Rheims damaged, and Arras almost destroyed. The French army was pushed back to within forty miles of Paris. It was only through a massive effort

A.S. Travis, *The Synthetic Nitrogen Industry in World War I*,
SpringerBriefs in History of Chemistry, DOI 10.1007/978-3-319-19357-1_1

during September 6–8 that the German advance was brought to a halt at the First Battle of the Marne by an Allied counter-attack on a front extending from Verdun to the Oise. The Germans retreated a short way to the north across the River Aisne. Both sides then raced north to the coast. The Kaiser's army gained the ridge that overlooked Flanders, at the First Battle of Ypres (October–November). The battlefront soon stretched four-hundred-and-forty miles, from Dunkirk, across Belgium and northern France, to the Swiss border. The subsequent stalemate on what became known as the Western Front would last almost four years.

All belligerents were unprepared for the prolonged war; hardly any effort had been made to mobilize industry in a support role. Only a few, and mainly chemists, had considered the critical role of chemical industry in the supply of modern explosives. In particular, the availability of nitrogen products was seriously underestimated by the over-optimistic German military. That changed with the stalemate that began on the banks of the River Marne, and ensuing systems of long, winding trenches excavated by both sides from early 1915. This stimulated new approaches to large-scale warfare based on nitrogen products, later capturing the public's attention in ways quite unlike that in which the very same or similar products, as fertilizers, would serve to sustain so much of the world's growing population. It represented a major step in the near-total reliance of warfare on science and technology, and brought on the rapid emergence of the first major modern military-academic-industrial complexes.

While the massive expansion of the nitrogen industry during World War I is a principal focus here, our historical understanding of how it was enabled to take place requires a thorough review of formative developments during the previous several decades. Here the story begins with the importation into Europe from the west coast of South America of naturally occurring nitrogen-containing products, guano (containing uric acid and other nitrogen compounds) and sodium nitrate, that enabled significant improvements in crop yields. This was a major enterprise from the 1840s, around which time agricultural science developed, with an emphasis on the role of fertilizers, and nitrogen-containing manures in particular. Over the following years, agriculture experiment stations opened, at first relying on the studies of one or two individuals, generally with training in chemistry. With the growing importance of the need to satisfy the food requirements of expanding populations, and the emergence of modern agricultural sectors, colleges of agriculture were established, in both Europe and the United States.

By 1880 the best quality guano had been consumed, and there were concerns in Europe over how long the supply of nitrates would last. This encouraged laboratory and industrial efforts to directly fix the unreactive gas nitrogen in the atmosphere as stable compounds that could be used as fertilizer. There were additional concerns, since from imported nitrate was obtained the nitric acid required in the manufacture of explosives, such as Alfred Nobel's dynamite. Meantime the production of illuminating gas, and later coke, from coal encouraged recovery of the byproduct ammonia for use as a fertilizer.

The widespread availability of electrical power during the 1890s directed the research effort towards fixing nitrogen, by its combination with oxygen, in the flame generated by an electric arc. This was useful in the manufacture of nitric acid required by the explosives industry, as well as of nitrate fertilizer. Another approach, also requiring electrical power, involved production of calcium cyanamide from calcium carbide. The solid cyanamide released ammonia when applied to the soil. These were the two main innovations of the first decade of the 20th century, and, provided that the supply of electrical power was cheap enough, offered some independence from the naturally occurring nitrates.

It was not only the finite reserves of Chilean nitrate that caused concern. The supply was controlled by commercial monopolies, which in turn also encouraged ongoing research into synthetic alternatives. This was particularly the case in Germany, a nation with limited natural resources, and dependent on the South American product to support its extensive agricultural industry. The research effort was further stimulated by the fact that the large German chemical corporations engaged in the manufacture of synthetic or coal tar dyes had begun to diversify, taking up interests in areas of chemistry other than those based on organic chemical products. Nitrogen fixation by its combination with hydrogen to give ammonia was attempted, in Germany and elsewhere, but each worker in turn abandoned the study when low yields, explosions, and other complications hindered the work. Then in 1909, at the Karlsruhe Technische Hochschule, a benchtop process was demonstrated in which nitrogen and hydrogen were combined at a high temperature and under pressure in the presence of a catalyst. By 1913 this had become the basis of an industrial process that was worked for production of the fertilizer ammonium sulphate. A novel process for manufacture of nitric acid was also taken up in Germany, the catalytic oxidation of ammonia from gas works, and increasingly from the new synthetic nitrogen processes. The nitric acid processes were not worked on a large scale before August 1914, and often in any case encountered technical difficulties in operation.

The military stalemate reached in September 1914 led to immediate and unprecedented demand for the nitrogen products ammonia and nitric acid, as well as for coal tar toluene and phenol (carbolic acid) that were nitrated, giving TNT (trinitrotoluene) and picric acid, respectively. That demand became even more urgent in Germany when at the end of the year, through the efforts of the British Royal Navy, German manufacturers of nitric acid and explosives were denied access to Chilean nitrate. From then on, the Kaiser's chemists and industrialists gave top priority to the capture of atmospheric nitrogen for both feeding the population of Germany and slaughtering the armies of the Entente Powers, the United Kingdom, France and Russia, generally referred to as the Allies (joined by Italy in 1915). This was the turning point in the modern manufacture of nitrogen products. State sponsorship of and industrial investment in essential nitrogen products enabled the construction of vast chemical works for purposes of war, but with the potential of no less vast markets after the war. German science and technology succeeded magnificently, something over which even the Allies were in agreement. Moreover, and as a measure of the complexities overcome by German scientists and technologists, their achievements could not be matched elsewhere. Fortunately for the Allied nations, they could continue to rely on the natural nitrates, despite submarine attacks on merchants shipping. In many respects, the impact during the years immediately following cessation of hostilities was no less great, as Germany's former enemies fought to catch up in nitrogen fixation processes and the development of high-pressure chemical technology that it spawned. No less than the synthetic dye industry, the synthetic nitrogen industry was seen as a key strategic sector that was essential to the survival of every industrialized, and industrializing, nation.

The main personality in this account of the success of large-scale fixation of atmospheric nitrogen is the German physical chemist Fritz Haber, who has been accorded the dubious honours of enabling Germany to conduct war after September 1914, and of introducing large-scale gas warfare in April 1915. Notwithstanding his ingenious solution to fixing atmospheric nitrogen, the former honour was, and is, based more on rhetoric and propaganda put out by industrialists and scientists to suit political masters in both England and Germany than was actually the case. More recently, and as the result of concerns of later generations, it has become the fashion to sensationalize Haber's role in these two chemistry-related sectors of modern warfare. Haber has come to personify the evil scientist who unquestionably places his personal interests and those of the state he serves above all else. For this reason, it is occasionally necessary here, for sake of completeness, to turn off the main route of the narrative to explore subsidiary themes related to Haber's activities.

Among other issues, I examine some purported, or at least "advertised," facts, including certain persistent myths concerning Haber's roles, and reinterpret the foundations of stories about the "Haber factories" that many believed almost alone enabled Germany to wage war from the autumn of 1914, or even earlier, right through to the Armistice on 11 November 1918. While these are shown to be erroneous, there is no denying that Haber's role as an outstanding administrator of war-related affairs during the "Chemist's War" were no less significant than his scientific and technical expertise.

1.2 Background to Fixed Nitrogen

The gas nitrogen constitutes about 80 % of our atmosphere. It is essential to plant growth, as fertilizer. There are few natural forms in which it is found in a combined state, for direct, or indirect, application. Formerly the principal supply was animal excrement, from which was obtained potassium nitrate (saltpetre, or nitre), also employed in gunpowder and the manufacture of nitric acid.

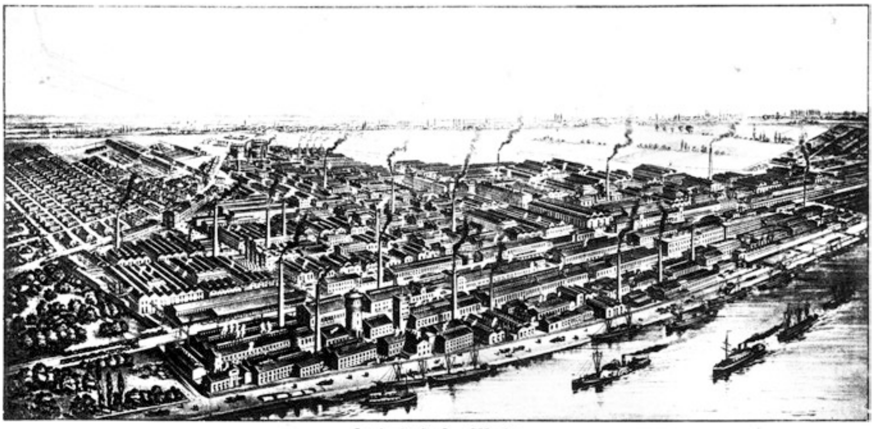
Attempts to combine atmospheric nitrogen with other elements, such as hydrogen to give ammonia, and oxygen to afford oxides which could be readily converted into nitric acid, reportedly formed the basis of 3,000 publications and patents by 1914. Mostly, they were unsuitable for commercial development, due to inefficiency of the methods, lack of scientific and technical knowhow, and high energy and installation costs.

Studies of nitrogen-containing gases go back to the last quarter of the 18th century. Joseph Priestley (1733–1804) was the first to examine the chemical composition of ammonia (NH_3), which in 1774 he decomposed with an electric spark. The colourless, alkaline gas, was known long before. Ammonia is named after the Temple of Jupiter Ammon in the oasis of Siwa in the western Egyptian desert [1]. It was there that, so the story goes, sal ammoniac (ammonium chloride) was found in camel dung and decomposing remains of animal sacrifices left out in the searing desert heat.

In 1772, Priestley established the composition of nitric oxide (NO, nitrogen monoxide), and also reported nitrous oxide (N_2O). In 1777 he prepared nitrogen dioxide (NO_2) [2]. Henry Cavendish (1731–1810) made nitric oxide using frictional electricity to spark together nitrogen and oxygen, and obtained what he called nitrous acid (nitric acid) [3]. Around the turn of the century Humphry Davy (1778–1829) prepared and analysed oxides of nitrogen [4]. The French chemist Frédéric Kuhlmann (1803–1881) in 1838 reacted nitric oxide with air to form nitrogen dioxide which with water afforded nitric acid [5]. This became the basis of a French patent filed early in 1859 by Madame Louise Lefêbvre. (Her English patent was dated 26 April 1859.) Unfortunately theories of physical chemistry were not advanced sufficiently to assist her, nor was there an economical source of electricity to bring about the continuous combination of oxygen with nitrogen.

An alternative approach used the combination of metals with nitrogen at elevated temperatures to form metal nitrides, which in turn yielded ammonia by reaction with steam. This was the approach taken in France during 1859 by Charles Tellier (1828–1913), who in 1880 formed a short-lived company to license this route. Ottokar Serpek in Norway worked with nitrides of aluminium, before devoting his firm's activities more profitably to extraction of aluminium.

The more viable processes for fixing atmospheric nitrogen, those described in this monograph, had to wait until the start of the 20th century. Of the three main rivals, it was the Haber-Bosch process, inaugurated in 1913 by the German dye-making firm Badische Anilin- & Soda-Fabrik (BASF, also once known as the Badische) of Ludwigshafen, almost opposite Mannheim, on the west bank of the River Rhine, that would come to the fore by the end of World War I (Fig. 1.1).



Ludwigshafen Works.

Fig. 1.1 BASF factory, Ludwigshafen, 1890s. (Sidney M. Edelstein Library)

BASF and the other leading German dye firms were founded in the 1860s and 1870s. In 1904 they formed two groups of loose alliances: BASF, AGFA (Actien-Gesellschaft für Anilin-Fabrikation), and Bayer, together known as the Dreibund, or Triple Confederation; and Leopold Cassella & Co. and Hoechst, the Double Alliance, in 1907 joined by Kalle & Co., and from then on known as the Tripartite Association. In 1916, at the height of World War I, the two groups formed a community of interests, the Interessengemeinschaft der deutschen Teerfarbenindustrie, often called the I.G. In 1925, they merged to create the behemoth Interessengemeinschaft Farbenindustrie Aktiengesellschaft, better known as I.G. Farbenindustrie AG, or I.G. Farben.

1.3 Natural Nitrogen Fertilizers

For much of the 19th century, the principal source of fixed nitrogen for use in agriculture and the manufacture of nitric acid was the west coast of South America. First was a natural fertilizer, guano, found in great abundance mainly on the three Chincha Islands, off the coast of Peru, close to the town of Pisco (south of Lima). Its history, in Europe at least, began after the explorer and geographer Alexander Humboldt (1769–1859) and botanist Aimé Bonpland (1773–1858) visited Spain's American colonies. During five years, from 1799 to 1804, they gathered information on geology, botany, anthropology, and economics, as well as specimens of plants and minerals. One of the latter was a substance covering the three islands, with the power, as Humboldt was informed by the Spanish, who learned it from the Indians, of greatly aiding the growth of plants. This was guano. On return to Paris, where Humboldt was residing, he communicated with the chemists Antoine-François Fourcroy (1755–1809) and Louis-Nicolas Vauquelin (1763–1829), requesting an analysis of guano. They had just completed an analysis of bird excrement, which Humboldt believed, correctly, had a composition similar to that of guano. In 1805 the two chemists published their analysis of Humboldt's sample, demonstrating its nitrogen content: the Chincha Islands were comprised of vast amounts of fixed nitrogen (mainly uric acid) in the form of bird droppings. Guano had also accumulated in many places along the coastline of mainland Peru.

This was less than a decade after the English cleric Thomas Robert Malthus (1766–1834) in his *An Essay on the Principle of Population* warned that food supplies could not keep pace with population growth. The time was ripe for sending guano to Europe. Nevertheless, it took until 1840 before guano was first exported, to England, from where the European market was mainly financed and controlled. Less than 2,000 tons arrived in 1841, but within a decade this figure had risen to over 200,000 tons per year. The Caribbean Island of Rodonda was also a significant source of guano, from 1843 until just after the outbreak of World War I (from the mid-1860s it was the inspiration for a mythical kingdom). Americans also took a particular interest in guano. In 1856 Congress passed the Guano Islands Act that empowered US citizens to take possession of unoccupied islands containing guano.

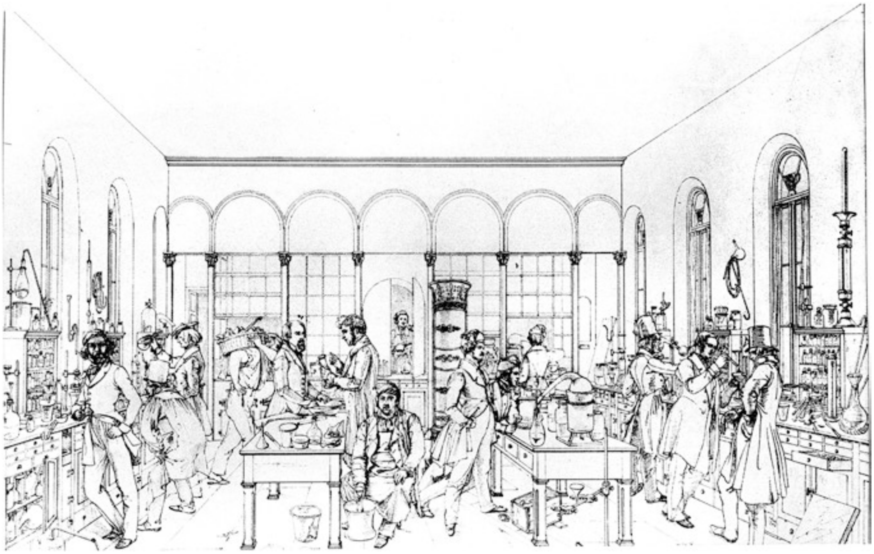


Fig. 1.2 Justus Liebig's laboratory at Giessen, around 1840. Liebig was ennobled in 1845. From J. Hofmann, *Acht Tafeln zur Beschreibung des chemischen Laboratoriums zu Geissen*. Heidelberg, 1842. Lithograph by Trautschold and Ritgen. (Sidney M. Edelstein Library)

The role of excrement, human and animal, as well as waste from crop production, rather than guano, as sources of fertilizer on arable land, was emphasized by various individuals, including John Mechi (1802–1880), in England in the 1840s, and the German chemist Justus Liebig (1803–1873), at Giessen (Fig. 1.2) [6]. Liebig, the leading promoter of agricultural chemistry, was for some time uncertain of the value of added nitrogen in the form of guano, at least until its phosphate content was established.

1.4 Fertilizers and Agriculture Experiment Stations

The scientific approach to agriculture, with a major input from chemistry, relied on agriculture experiment, or research, stations, dedicated colleges, and associations of agriculturalists. Their histories in Europe went back to the 1830s and early 1840s. In France during 1836, Jean-Baptiste Boussingault (1802–1887) opened a research station some 60 km north of Strasbourg, where his experiments included studies on the role of nitrogen in plant growth. Boussingault's work would be brought to an abrupt halt at the outbreak of the Franco-Prussian War of 1870–1871. In 1842, James F. W. Johnston founded the Agricultural Chemistry Association of Scotland. One year later John Bennett Lawes (1841–1900) began field experiments at Rothamsted, Harpenden, north of St. Albans, England, in partnership with the chemist Joseph Henry Gilbert (1817–1901) who emphasized the